Polymer Science at the NIST Combinatorial Methods Center

Levels of Participation

Participating Membership
Focused Projects Membership
Partner Membership

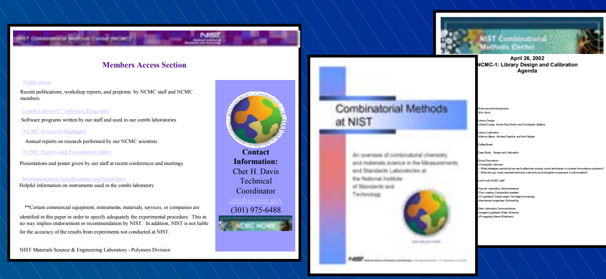
*Membership levels based on different
Degrees of project involvement
Please visit www.nist.gov/combi for more details

A consortium of industry and national laboratories

Member Benefits

- Attendance at NCMC bi-annual member meetings featuring presentations from NIST scientists, invited academic speakers, informative poster sessions, hands-on laboratory demonstrations, and member panel discussions.

- Access to a Member Only section of the NCMC website containing publications from NIST scientists and NCMC members, instrumentation specifications and guidelines, posters and presentations from recent conferences and meetings, research highlights, and software programs



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Combinatorial Informatics System

W. Zhang, M. Fasolka

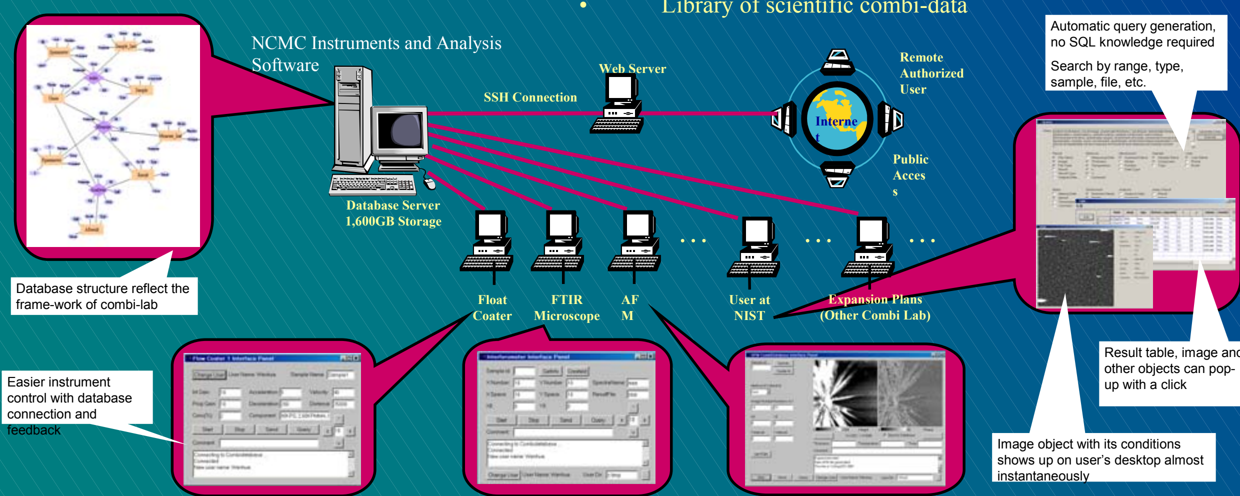
Progress

- Database system selection
- Database server setup
- Security / data protection issues
- Basic framework of the database
- Backbone interface programming

CTCMS

Informatics & feedback for:

- Data management, integration
- Data visualization
- Automated data entry, retrieval
- Protocols for standardized data formats
- Link to and refine other Combi processes formats
- Library of scientific combi-data

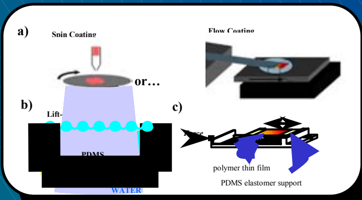


Cher H. Davis, Kathryn L. Beers, Aaron M. Forster, Christopher M. Stafford, Archie P. Smith, Christopher K. Harrison, Wenhua Zhang, Alamgir Karim, and Eric J. Amis

National Institute of Standards and Technology

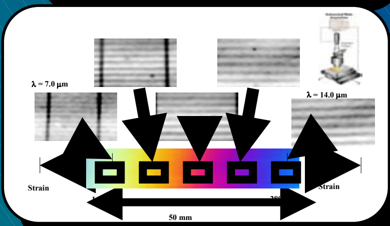
Strain Induced Elastomer Buckling Instability for Mechanical Measurements

C. Harrison, C. Stafford



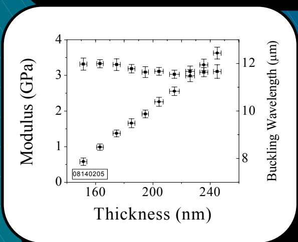
Sample Preparation

Thin polymer films are fabricated via spin or flow coating on a silicon wafer. The thickness is measured by conventional interferomtery. The polymer film is transferred onto a clear silicon sheet (polydimethylsiloxane, PDMS) by aqueous immersion. Upon straining, the bilayer undergoes a mechanical instability with a well-defined wavelength. The micron-scale wrinkles produce the iridescent sample shown above.



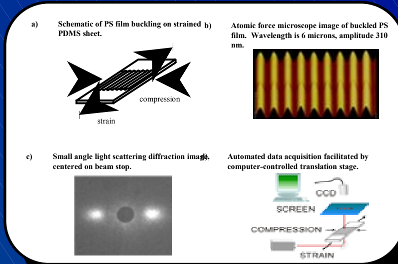
Buckling of Films with a Thickness Gradient

Flow coating was utilized to fabricate a PS film with a thickness gradient ranging from 140 to 280 nm across a 50 mm sample. The polymer film was transferred to a PDMS sheet and the entire sample was strained. The buckling wavelength ranges from 7.0 to 14 microns (as shown on left). Each region can be treated as an independent sample as SIEBIMM is a local measurement with a minimum sample size dictated only by the laser beam diameter, typically 0.5 mm or less. The vertical lines are cracks.



SIEBIMM Measurements on PS Films with a Thickness Gradient

We use SIEBIMM to measure the modulus of a PS film with a thickness gradient created by flow coating. The calculated modulus is 3.2 +/-0.1 Gpa, markedly independent of thickness. Thickness effects on the measured modulus should begin to play a role for films of thickness comparable to the polymer's radius of gyration, *c.a.* 10 nm.

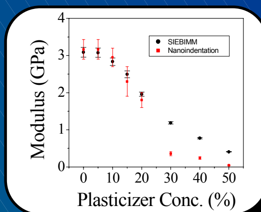


SIEBIMM Measurements on Polystyrene Films

We use a 100 nm thick polystyrene (PS) film to demonstrate SIEBIMM. Straining the sample induces the mechanical instability. Light scattering is used to rapidly measure the wavelength with computer controlled strain and translation stages. The modulus of the film, E_p, can be calculated by the following equation, which requires the modulus of the PDMS (E_m), the Poisson ratio of the film (υ_p), the buckling wavenumber q and film thickness h.

$$E_p = \left(\frac{12E_m}{(3-\upsilon_p)(1+\upsilon_p)}\right)[qh]^{-3}$$

We measure a modulus of approximately 3 Gpa, in good agreement with published values for PS.



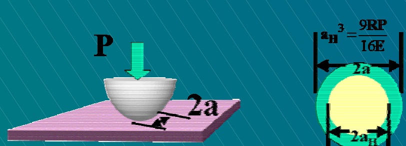
SIEBIMM on Plasticized Films

Thin films were spin coated from a blend of PS with a commonly-used plasticizer, dioctyl phthalate (DOP). The measured modulus is shown to decrease in a sigmoidal fashion with the loading fraction of DOP. Approximately forty measurements are made per sample (total time: 4 minutes) and the error bars are generated from the scatter in the measurements. We are no beginning to apply this technique to plasticized poly(vinyl chloride).

Multi-lens Combi Adhesion Tests

A. Forster, C. Stafford

The Johnson, Kendall, Roberts (JKR) theory provides quantitative information about the adhesion forces exerted between two contacting materials. The Multi-lens Combinatorial Adhesion Test (MCAT) is a high-throughput JKR technique under development to investigate adhesion forces across combinatorial libraries.



$$a_H^3 = \frac{9RP}{16E}$$

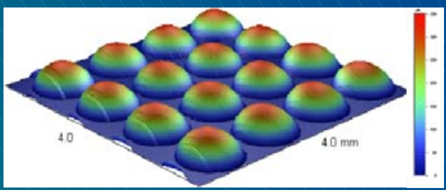
The JKR theory relates the contact area between a sphere and flat substrate to the applied load as a function of the modulus, radius-of-curvature of the indenter, and the work of adhesion.

Experimentally, load (P) or lens displacement (d), radius-of-curvature, and contact area (a) are measured to determine the work of adhesion (G) and the modulus (E).

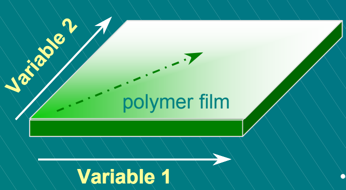
JKR Equation

$$a^3 = \frac{9R}{16E}\left[P+3\pi GR+\sqrt{6GRP+(3\pi GR)^2}\right]$$

In a traditional JKR test, a single, large spherical indenter is used against a flat substrate. This multi-lens technique utilizes an array of smaller lenses to perform hundreds of individual JKR experiments across an area of 1 to 10 cm^2, during one loading and unloading cycle.



The multi-lens array lends itself to a wide variety of experimental scenarios. This flexibility aids in the design of data libraries for adhesion studies.



Combinatorial Library

Utilizing polydimethylsiloxane (PDMS) molds the lenses may be fabricated from:

- *commercial adhesives*
- *Glass*
- *epoxy*
- *PDMS*

The flat substrate may possess gradients in:

- *Roughness*
- *surface energy*
- *composition*
- *thickness*

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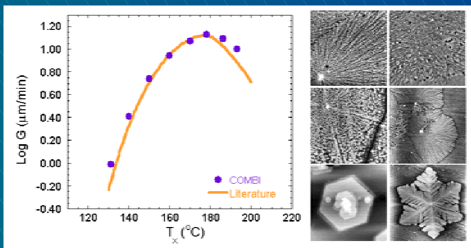
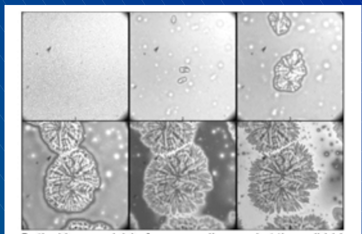
Polymer Crystallization

K. Beers, M. Walker, H. Wang, J. Douglas

Developing combinatorial and high throughput characterization methods for polymer crystallization in thin films. High speed automated scanning techniques such as optical, and surface force microscopy and light scattering can be used for evaluation of growth rate and other properties under orthogonally varying process variables.



Growth rate (G) as a function of temperature for isotactic polystyrene crystallized on a temperature gradient (left) and examples of crystalline superstructures obtained across the temperature / film thickness surface map.

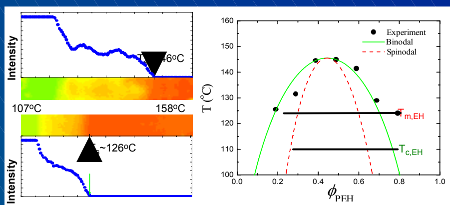


Optical images (a) before annealing; and at times (b)14; (c) 39; (d) 69; (e) 99; and (f) 129 minutes after placement on gradient temperature stage for isotactic polypropylene nucleated with 2-biphenyl carboxylic acid.

- Additive Composition Gradients: the effects of nucleating agents on kinetics and morphology in isotactic polypropylene

- Validation studies on model systems: mapping kinetics and morphology in isotactic polystyrene

- Blend Composition Gradients: mapping phase diagrams for blends of polyethylene copolymers that undergo both phase separation and crystallization



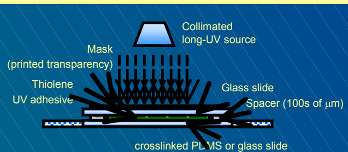
Intensity of scattered light as a function of temperature for blends annealed on gradient temperature stage (left) and phase diagram for the PEH/PEB blend system (right).

Polymer Formulations

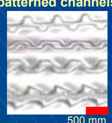
K. Beers, H. J. Walls, J. Cabral, S. Hudson

Project Goals

- Objective: To develop HT or combinatorial methods for measuring properties, such as viscosity, interfacial tension, wettability, compatibility and reactivity, of polymeric mixtures involving multiple component types
- Problem: How to design systematic approaches to measure the many complex, poorly understood interactions presently addressed with little more than empirical knowledge?
- Approach: Develop *modular, rapid and small scale* fluidic reactors, mixing devices and measurement techniques
- Focus: Emulsions and polymeric surfactants



Topographically patterned channels



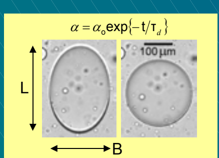
Design and Fabrication

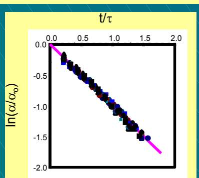
Rapid prototyping technique for fabrication of fluidic channels in a solvent-resistant polymeric matrix

- Conventional contact lithography
- Commercially available thiolene-based adhesive
- Matrix can be crosslinked UV curable adhesive and glass (solvent resistant) or, be transferred to PDMS and sealed against glass (aqueous applications)
- Both result in optically transparent, sealed, micro/millimetric fluid handling devices.

Interfacial Tension (ITF) Focused Project

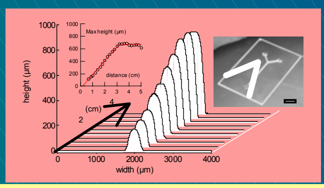
- Non-proprietary projects on new methods and applications in areas of interest to participants
- Prioritize development of milli-fluidic instrumentation for rapid IFT measurements
- Droplet deformation under pressure driven flow fields
- Measuring relaxation of deformed droplets in fluidic channels while continuously varying droplet and matrix composition
- Compliment related viscosity and stability measurements

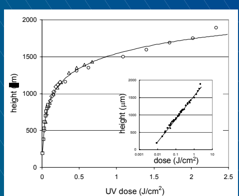




Structural Control and Characterization

- Control the lateral dimensions (determined by mask & collimation)
- Control vertical dimensions (determined by UV dose and, ultimately, by the spacer)





height= α ln(UVdose) + β

α,β=f(adhesive)

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